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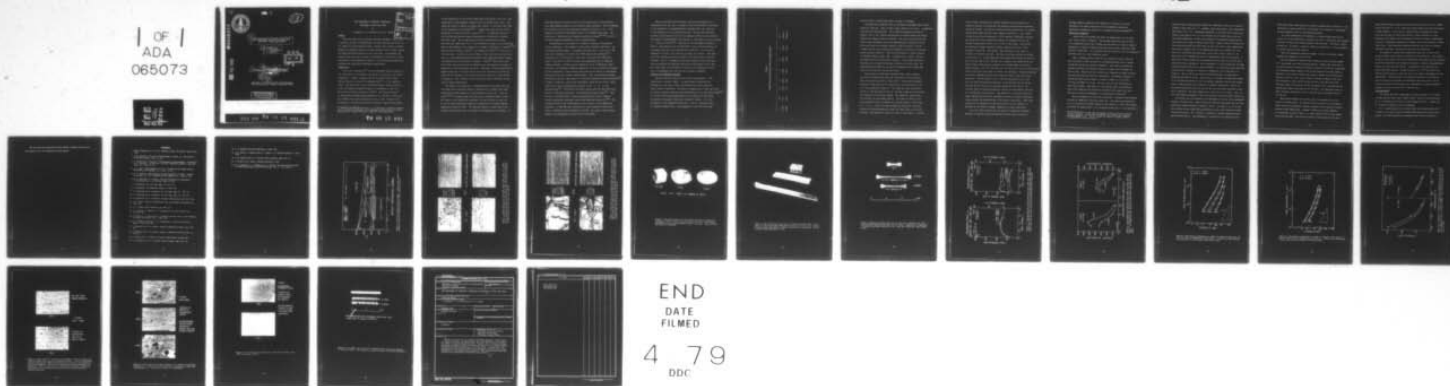NOV 78  J WADSWORTH, L E EISELSTEIN                     N00014-75-C-0662

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(6) THE DEVELOPMENT OF ULTRAFINE, SUPERPLASTIC
STRUCTURES IN WHITE CAST IRONS.

(10) J. Wadsworth
L. E. Eiselstein
O. D. Sherby

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Department of Materials Science and Engineering
Stanford University, Stanford, California 94305

# THE DEVELOPMENT OF ULTRAFINE, SUPERPLASTIC
# STRUCTURES IN WHITE CAST IRONS

by

J. Wadsworth, L. E. Eiselstein and O. D. Sherby[*]

## SUMMARY

White cast irons are not normally considered malleable. Recent work at Stanford, however, has demonstrated that these materials can in fact be hot forged, hot rolled and warm rolled. Further, such a series of thermo-mechanical treatments refines the as-cast structure so as to impart unusual and highly beneficial properties to the cast irons. In particular, the worked, white cast irons are found to be superplastic and there is a great improvement in room temperature properties. This processing procedure may therefore have significant technological applications.

## INTRODUCTION

A cast iron is defined as[1] "an iron containing carbon in excess of the solubilities in the austenite that exists in the alloy at the eutectic temperature". Further, "a white cast iron is one that gives a white fracture because the carbon is in combined form"[1], i.e. as cementite ($Fe_3C$). This means that an alloy containing carbon in excess of point E, in the iron-cementite diagram of Figure 1, is defined as a cast iron. This point is correctly located at 2.11%C[1]. Also shown on Figure 1 is the generally accepted phase diagram prior to ~1950 and it can be seen that formerly this point was believed to be at ~1.7%C. Because of the discrepancy in the reported location of this point, some confusion exists

*J. Wadsworth is Research Associate, L. E. Eiselstein is Research Assistant and O. D. Sherby is Professor in the Department of Materials Science and Engineering at Stanford University, Stanford, California 94305.

in the literature as to the correct carbon level that defines a cast iron. The significance of this apparently semantic point will become obvious later. One thing that nearly all authors are agreed upon, however, is that white cast irons are not considered to be malleable[2-6]. Indeed, early texts used the lack of forgeability of white cast iron to define it[2-4]. In consequence little, if any, work has been directed toward investigating the feasibility of working white cast irons. This is especially the case involving working of white cast irons that remain white (i.e., do not graphitize) during or after processing. There is some evidence in the literature of attempts by Piwowarsky, in Germany[7-11], which has been reviewed by Grant[12], and attempts by Forbes[13] in the U.S.A. to roll cast irons. In nearly all cases, however, these were either not white cast irons, or, they graphitized during processing thereby undergoing radical changes in properties due to the presence of graphite. Forbes[13] does claim to have rolled white cast iron which did not graphitize but unfortunately no details of temperature or rolling procedure are given. Indications in these publications do suggest though that if rolling could be carried out successfully in white cast irons that the refinement of the resulting structure would be of great benefit to properties. Also, a product such as thin sheet, which is difficult to cast, could be readily manufactured.

Our belief in the feasibility of thermomechanically processing white cast iron stems from an extensive program of research into ultrahigh carbon (UHC) steels which has been underway at Stanford University since 1973. This research[14-19], has demonstrated that such steels, containing between ~1% and 2%C, Figure 1, and traditionally believed to be very difficult to work, can in fact be readily worked and unusual warm formability and excellent ambient temperature properties result. The important feature of these steels is the ultrafine microstructure that is developed by thermomechanical processing. In

2.

these UHC steels the as-cast structure is the expected one of coarse pearlite
with large cementite plates at prior-austenite grain boundaries. After processing,
the structure is one of fine spheroidized cementite particles of ~0.1 - 0.5μm
diameter in a matrix of fine-grained ferrite (~1μm diameter grain size). The
properties of these steels are discussed extensively elsewhere[14-19] but one of
the major properties developed is that of superplasticity.

Superplasticity is the ability of a material to deform to extraordinary
tensile elongations. This property is only found in materials which have - and
can maintain during forming - an extremely fine grain size. In the case of UHC
steels this elongation can be well over 1000% under optimum conditions[17].
Superplasticity in these steels is found over a relatively wide temperature
range of about 600°C to 900°C at strain rates approaching those of commercial
forming operations. Although tensile elongation is used to demonstrate super-
plasticity, the significant commercial parameters are the low stresses of forming,
the low temperatures at which forming takes place and the ability of superplastic
materials to flow into extremely complex shapes. Thus one superplastic forming
operation may replace a large number of traditional forging and joining operations[20].
A number of commercial, non-ferrous, superplastic alloys are now in use. Super-
plasticity is used in manufacturing components in Ni, Ti, Cu, Al, and
Zn based alloys[21]. Particularly successful applications have been found using
IN-100, a nickel-based superalloy, for manufacturing turbine blades[22]. Also,
the use of a Ti-6Al-4V alloy, formed superplastically, has found a number of
aircraft applications including a Nacelle beam frame and intake ducts[23]. The
ultrafine structure that is developed is also of benefit to room temperature
properties. A 1.6%C UHC steel typically has a yield strength of 120-140 ksi,
a UTS of 160-180 ksi, and an elongation of 10-20%. The range of values reflect
whether or not annealing is carried out after processing.

Based on experience with UHC steels, and the obvious benefits of a superplastic white cast iron, a program of study was undertaken to determine 1) the feasibility of thermomechanically processing white cast irons and 2) the properties of such processed materials. The potential additional problem in processing white cast iron as opposed to UHC steel is as follows. The methods of processing UHC steels involve, at some stage, that all the carbon is taken into solution[16]. Referring to the iron-cementite diagram of Figure 1, and bearing in mind the definition of cast iron given earlier, it is apparent that this is not possible with a cast iron. At the eutectic temperature of 1147°C, only 2.11%C can be taken into solution. This means that coarse carbide particles will remain in the matrix after a solution treatment. This is a fundamental distinction between UHC steels and cast irons and presents the additional problem in the processing of cast irons. This upper limit of the solubility of carbon at any temperature in cast iron is a fundamental point and one that is worthy of emphasis.

## MATERIALS AND PROCESSING PROCEDURE

Two cast irons were chosen for this initial investigation . Their compositions are given in Table I. As may be seen one material just qualifies as a cast iron (2.13%C) and the other one is well within the limit (2.36%C) of definition. The choice of compositions of these cast irons was based on previous experience with graphitization in the iron-carbon system[18]. In the case of UHC steels, graphitization can be deleterious both during processing and subsequent superplastic forming or testing. It is also detrimental to room temperature properties. Since it is desirable then to eliminate graphitization, a high manganese (~1.5%) level was chosen

4.

## Table I

### Compositions of White Cast Irons by Weight Percent

| C | Mn | Si | P | S | Cr | V | Fe |
|---|----|----|----|----|----|----|----|
| 2.13%C | 1.41 | 0.08 | 0.018 | 0.008 | 0.032 | 0.038 | Balance |
| 2.36%C | 1.48 | 0.10 | 0.019 | 0.007 | 0.038 | 0.022 | Balance |

5.

and the silicon, a graphitizing agent, was kept to a minimum.

The ingots were supplied as 100 lb. castings of approximate square cross section of 76mm side. Cubes of 76mm x 76mm x 76mm were cut from the castings and these were the starting pieces. The as-cast microstructure is shown in Figures 2 and 3 and a typical microstructure of coarse pearlite and coarse proeutectoid cementite plates is observed. One cube of each cast-iron was used in these experiments and was thermomechanically processed as follows. The as-cast cubes were solution treated at 1120°C for about 2 1/2 hrs., A large fraction of the cementite was dissolved at this point to form high carbon austenite (Figure 1). They were then forged in a single pressing to "pancake" shapes of about 38mm in height and air cooled. The cube and forged pieces are illustrated in Figure 4. The forging presented no problems and no cracking was observed in either of the cast irons. The forging merely processes the casting into manageable form although some break up of the as-cast structure does occur.

Oblong blocks of approximate dimensions 38mm x 38mm x 76mm were cut from the "pancakes" and these were processed in the following way. First the blocks were heated to about 1100°C. This solution treatment dissolves a large amount of the cementite but as stated before not all of it can be dissolved (Figure 1). The blocks were then continuously rolled at 5-10% reduction per pass on a two-high, 178mm mill during which the temperature of the blocks dropped. The rolling was stopped when a dull red heat had been reached (650°C). The number of passes at this stage was about 16 and a thickness of 11.4mm was reached. (True strain of $\epsilon = \sim -1.2$). Little or no edge cracking was observed at this stage. During this rolling procedure two events are taking place in the microstructure. The material at 1100°C is in the $\gamma + Fe_3C$ region. As cooling

6.

occurs, carbon precipitates on existing cementite and also nucleates new cementite particles on dislocations and austenite grain boundaries generated during the rolling. The process of working during cooling results in a greatly refined matrix grain size and a fine cementite distribution. Upon cooling through the $A_1$ critical temperature ($\sim 727°C$) austenite transforms to pearlite. The second step of the process then consists of isothermally rolling the partially worked slabs at 650°C, i.e. in the $\alpha + Fe_3C$ range. This rolling was carried out using a reduction of about 5-10% per pass until a final thickness of about 3mm was reached. This represents a true strain, $\varepsilon$, of about -1.2 in about 12 passes. The total true strain involved in rolling is then about -2.4. During isothermal rolling some edge cracking was observed, more so in the case of the 2.36% cast iron. However, the cracks did not propagate during rolling and were confined to the extreme edges of the strip. Shown in Figure 5, for the 2.13%C cast-iron, is the block cut from the forged piece, and the strip after both stages of rolling. The isothermal rolling spheroidizes the pearlite remaining from the first stage and further refines the ferrite grain size. Shown in Figures 2 and 3 is the microstructure of the 2.13%C cast iron after processing and comparison can be made both with the as-cast structure and with the typical structures developed in UHC steels. The structure after hot and warm working now consists of fine cementite particles in a fine-grained, ferrite matrix. Also apparent in the microstructure are larger cementite particles that presumably are from the original undissolved carbide present from solidification at the eutectic temperature. These large carbides are not present in the UHC steels because all the cementite is dissolved at the solution treatment temperature in these compositions.

It is this final structure that was tested for mechanical properties. Specimens for tensile testing were machined from the final rolled strip.

The gage length was machined in the direction of rolling in all cases. Specimens of 1/2" gage length were used for superplastic evaluation. Superplastic testing was carried out on equipment described elsewhere[24].

RESULTS AND DISCUSSION

Typical tests to determine the extent of superplasticity in the white cast irons are shown in Figure 6. The specimens shown were tested at 650°C at an engineering strain rate of 1%/min and the results are shown in Figure 6. Elongations to failure of 526% and 291% were found for the 2.13%C and 2.36%C specimens respectively. For a material formerly believed to have no malleability whatsoever this is a truly remarkable result.

Shown in Figures 7a and 7b are the true stress-true strain curves for both cast irons at 650°C and 800°C at an engineering strain rate of 1%/min in both the as-cast and processed conditions. Apart from the enormous increase in ductility the dramatic decrease in strength after refinement is also of note. Studies were also carried out to determine the range of strain rate over which the cast irons are superplastic. The maximum elongation to failure was found at about 1%/min, as shown in Figure 8, at both 650°C ($\alpha$ + $Fe_3C$) and 800°C ($\gamma$ + $Fe_3C$). The cast irons are more superplastic at 650°C than at 800°C. This result is to be expected since at temperatures above 727°C cementite will dissolve and austenite grain growth will occur. This will of course be detrimental to the superplastic properties*.

Tests to determine the strain rate sensitivity, a parameter which measures the ease of formability, were also carried out. The strain-rate sensitivity, m, is to be found in the high temperature flow equation, $\sigma = K\dot{\varepsilon}^m$, which relates the stress for plastic flow, $\sigma$, to the applied strain rate, $\dot{\varepsilon}$, via a constant, K.

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* It is of interest to note that the reverse is true for the irons in the as-cast condition. In this case, elongations are improved and strength decreased <u>above</u> the $A_1$. This is because the removal of <u>coarse</u> cementite is beneficial to tensile ductility.

Perfectly plastic materials which behave like a Newtonian fluid (such as glass or molasses), have values of $m = 1$. Normally, metals and alloys at warm temperatures have values of $m = \sim 0.2$. Superplastic materials, on the other hand, usually have values in the range of $0.4 < m < 0.6$ and these are the values found in the cast irons investigated here. Strain rate sensitivity is determined by measuring the flow stress over a range of imposed strain rates. Figures 9 and 10 illustrate the flow stress variations with strain rate for both cast irons at three temperatures below the $A_1$ (Figure 9) and at two temperatures above the $A_1$ (Figure 10). The value of m, the strain rate sensitivity, can be seen to lie at about 0.5, for both irons, at all temperatures, over the superplastic range of strain rates. A value for activation energy can be determined from these data to be 203 KJ (48.5 Kcal) below the $A_1$ temperature. This corresponds to a value between that for grain boundary diffusion and that for lattice diffusion. By extracting further data from these two plots, strength as a function of temperature can be replotted as in Figure 11. Here it can be seen that the strength decreases as temperature is increased from 600°C to the $A_1$. Above the $A_1$, the strength increases both because of the decreased diffusivity of iron in austenite compared to ferrite and because there is some austenite grain growth due to cementite dissolution. This is an interesting observation since not only are the cast irons more superplastic below the $A_1$ temperature than above (Figure 8), but also they can be weaker. Thus, the cast irons exhibit strengths at 700°C that are lower even than at 800°C (Figure 11).

One feature that is disturbing is the appearance of cavities after superplastic testing in the cast irons. These cavities are invariably associated with the large undissolved carbides as shown in Figures 12 and 13. In Figure 12 it can be seen that cavities are not formed during processing but only after testing in tension at slow strain rates. The cavitation is observed at testing temperatures both above and below the $A_1$. This phenomenon of cavitation is not observed in UHC

steels where these large particles are absent as demonstrated in Figure 14. Such cavitation is clearly deleterious, both because of its influence on formability and its subsequent effect on post-forming properties.

As a final demonstration of formability, a simple forming operation was carried out on the superplastic, as-rolled, cast irons. Thin strips were deformed in torsion at 650°C until several complete turns were made. The result is shown in Figure 15. No surface cracking is visible in either iron. It is not possible to do this with the as-cast material.

These warm temperature properties suggest a break in traditional thought regarding the formability of cast irons.

The room temperature properties were assessed by tensile testing samples of 1" gage length on an Instron testing machine. Samples were tested both in the as-rolled and as-rolled plus annealed (at 650°C for 20 minutes) conditiions. For comparison purposes samples of the as-cast materials were also prepared. Materials in the as-cast condition were found to exhibit zero ductility and a fracture stress of 51 ksi. After rolling, the yield strengths of the irons were increased to about 130-150 ksi and the UTS to just above this level. Some small amount of ductility (~1%-2%) was usually found. Annealing did not significantly improve the properties of the irons. For compression tests on small cylinders, ductilities of up to 5-7% were found at strength levels of 125-150 ksi.

These results reflect the fact that some of the cementite is not as fine as in the UHC steels. The reason for this is that all of the cementite cannot initially be dissolved. However, cementite can be refined by further working at warm temperatures. Attempts to break up the large cementite by isothermally rolling at 770°C, i.e., above instead of below the $A_1$ temperature have been made. The irons were rolled in the same way for the first

10.

stage (working during cooling from 1100°C) and then rolled at 770°C to similar strains as before ($\varepsilon \simeq -1.2$). Both irons rolled readily with some small degree of edge cracking. In this case the rolled product is quenched into water after the last pass (to avoid the austenite → pearlite reaction) to form martensite plus cementite and then annealed at 650°C to produce a spheroidized structure. Superplastic properties are similar, after this procedure, to those found before. Room temperature properties show that the irons are stronger (typically yield point ~160 ksi) but still with low ductilities.

The problem we currently are attempting to solve then is that of producing an evenly sized cementite distribution of fine particles. This uniform structure should exhibit even better superplastic properties than those described herein while improving room temperature properties. Different thermomechanical methods are currently being studied. One approach not yet mentioned is a powder metallurgy approach. Already Caligiuri et al.,[25] have shown that fine-structured powders of white cast iron can be readily consolidated at warm temperatures. This method may be an appropriate approach to achieving the goal of uniform fine structures in superplastic white cast irons. We are currently involved in investigating the consolidation of rapidly solidified powders of a 2.4%C cast iron and a 3.0%C cast iron.

## ACKNOWLEDGEMENTS

The cast irons were supplied by Union Carbide of Niagara Falls and we are grateful to Dr. M. Korchynsky for this support.

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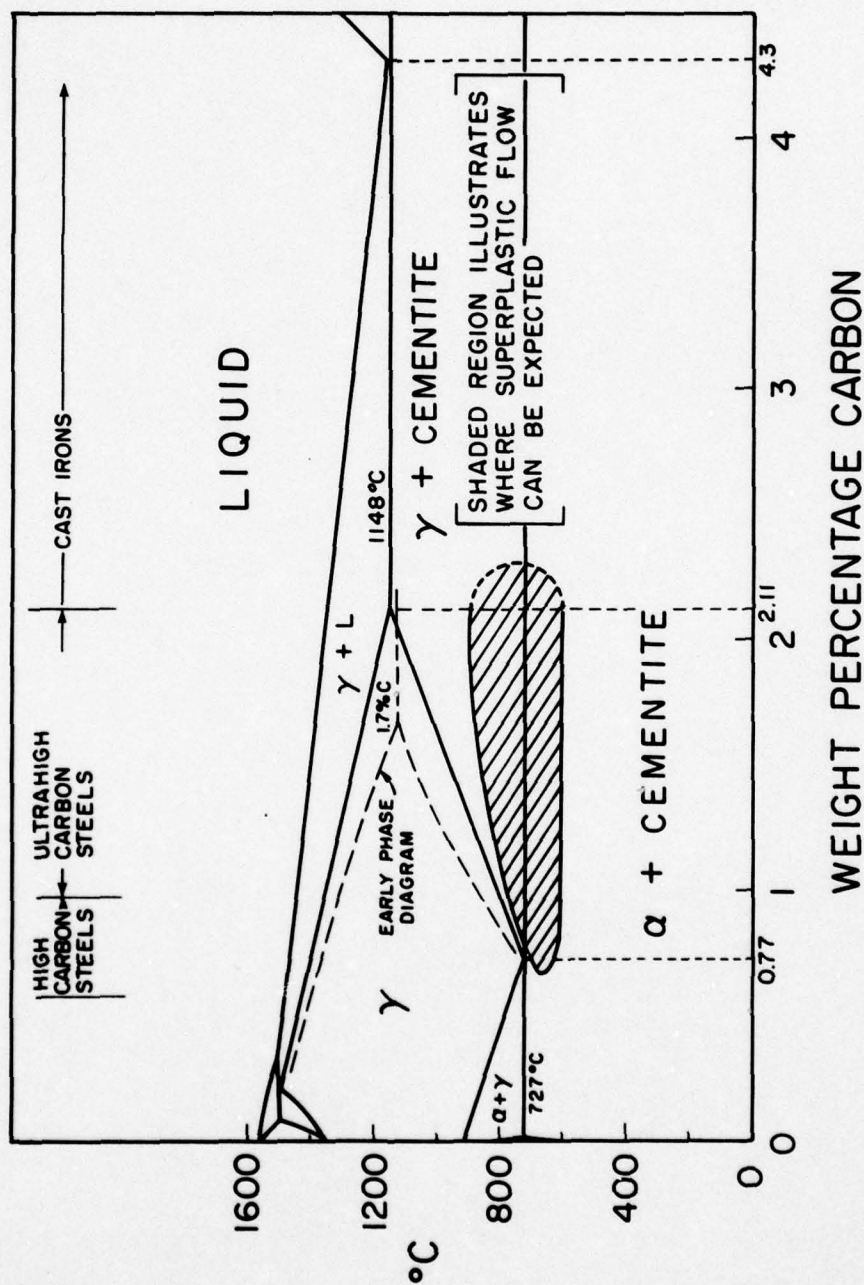
Figure 1. The iron-cementite phase diagram. Shown on the diagram are the generally accepted classifications of high carbon materials.

AFTER HOT AND WARM WORKING

ULTRA
-HIGH
CARBON
STEEL

100 μm
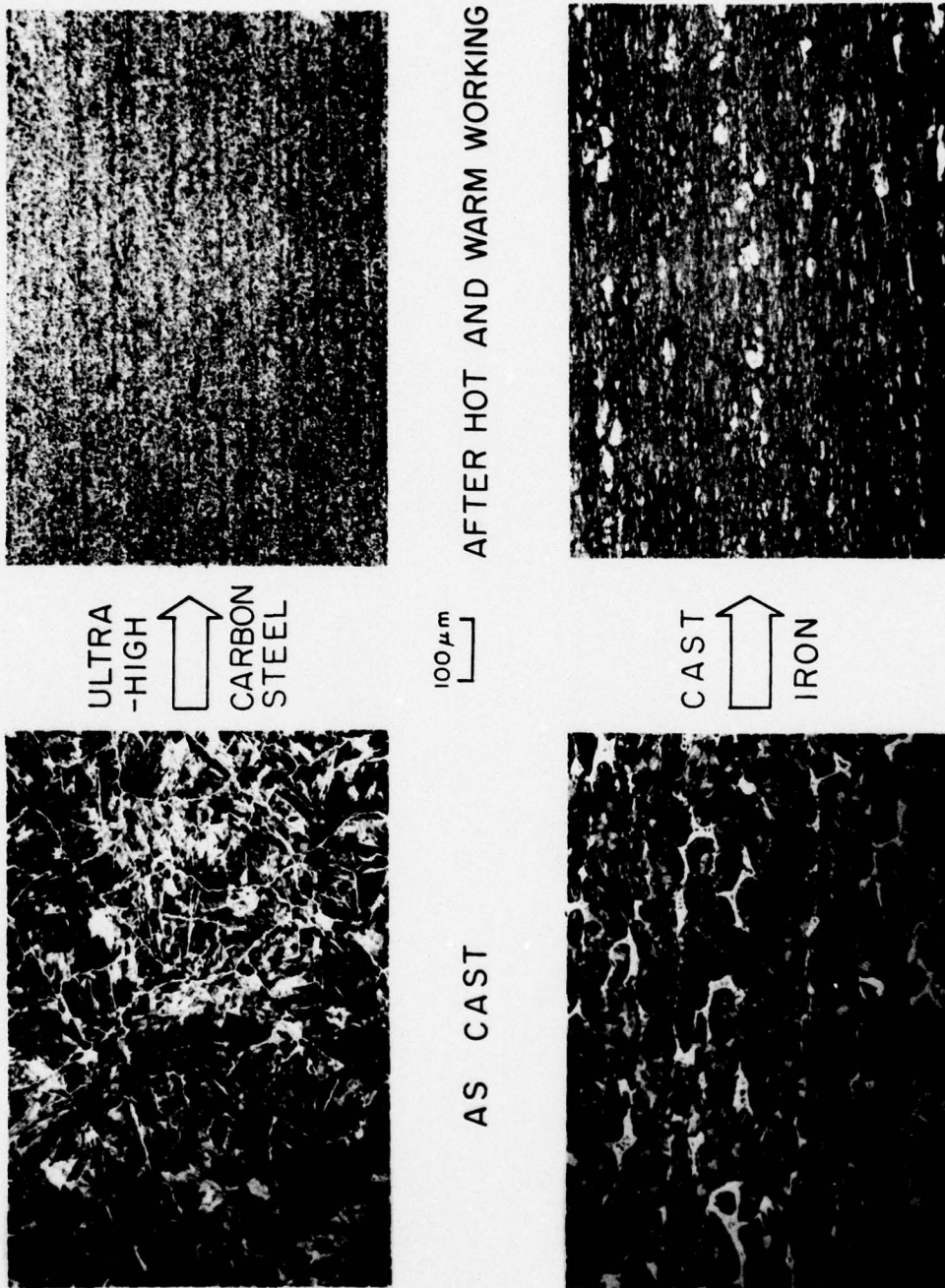
CAST
IRON

AS CAST

Figure 2. Shown above are an ultrahigh carbon (UHC) steel (of 1.6%C) and a cast iron (2.13%C) in both the as-cast and processed conditions. In the case of the cast-iron, it can be seen that large, undissolved carbides remain after hot and warm working.

16.

ULTRA -HIGH CARBON STEEL

CAST IRON

AFTER HOT AND WARM WORKING

10μm

AS CAST

Figure 3. Shown above are an ultrahigh carbon (UHC) steel (of 1.6%C) and a cast iron (2.13%C) in both the as-cast and processed conditions. In the case of the cast-iron, it can be seen that large, undissolved carbides remain after hot and warm working.
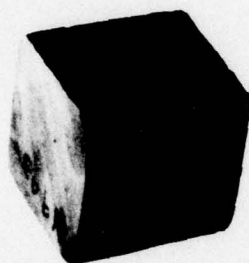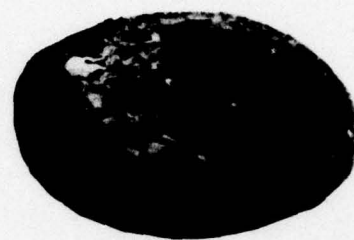
AS CAST                    2.13 % C                      2.36 % C

WHITE    CAST    IRONS    AS    FORGED    AT    1100°C

Figure 4. The first stage in the processing consists of single-step
pressing at 1100°C the as-cast block shown at the left of the above
photograph into the pancake shapes shown on the right.  Boron nitride
was used as a lubricant.

18.
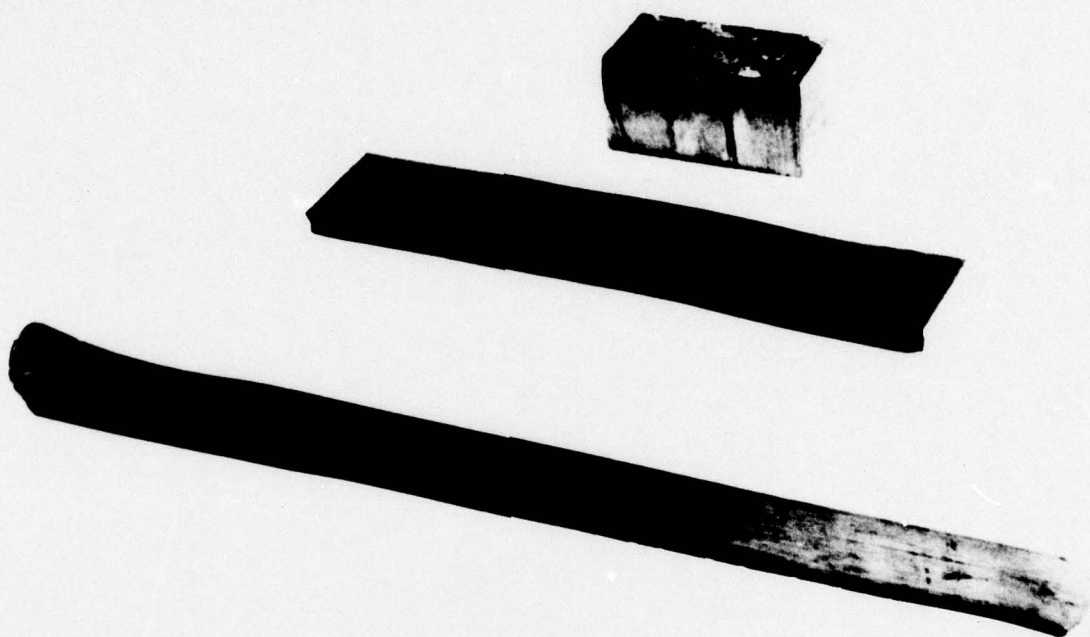
Figure 5. The 2.13%C cast iron after the three processing steps.  Top: a piece cut from the pressed shape shown in Figure 4. Center: after rolling during cooling from 1100°C to ~600°C, Bottom: after isothermally rolling at 650°C (final thickness ~0.12").
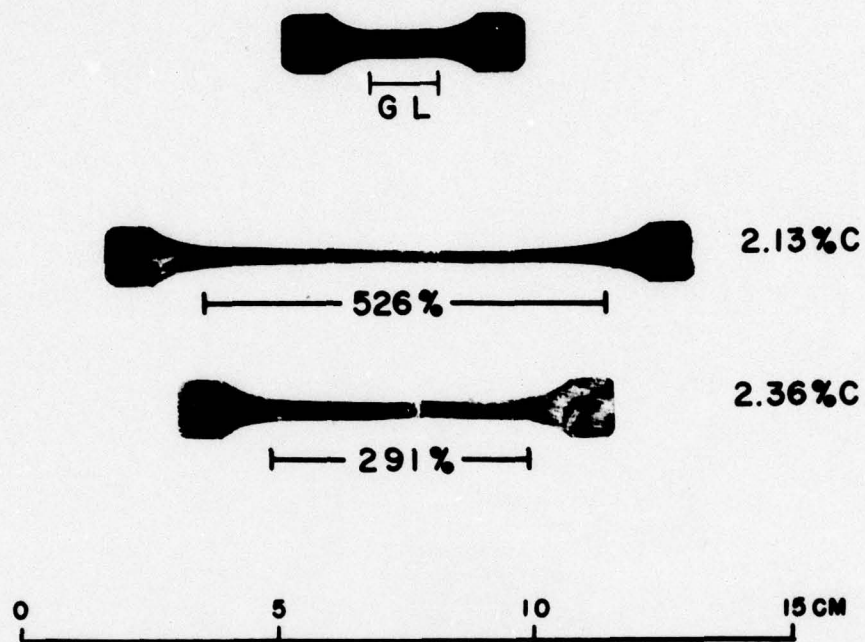
Figure 6. Results of tensile tests on the cast irons carried out at 650°C at an initial, engineering-strain rate of 1%/min. Superplastic elongations of 526% and 291% were recorded for the 2.13%C and 2.36%C cast irons respectively.
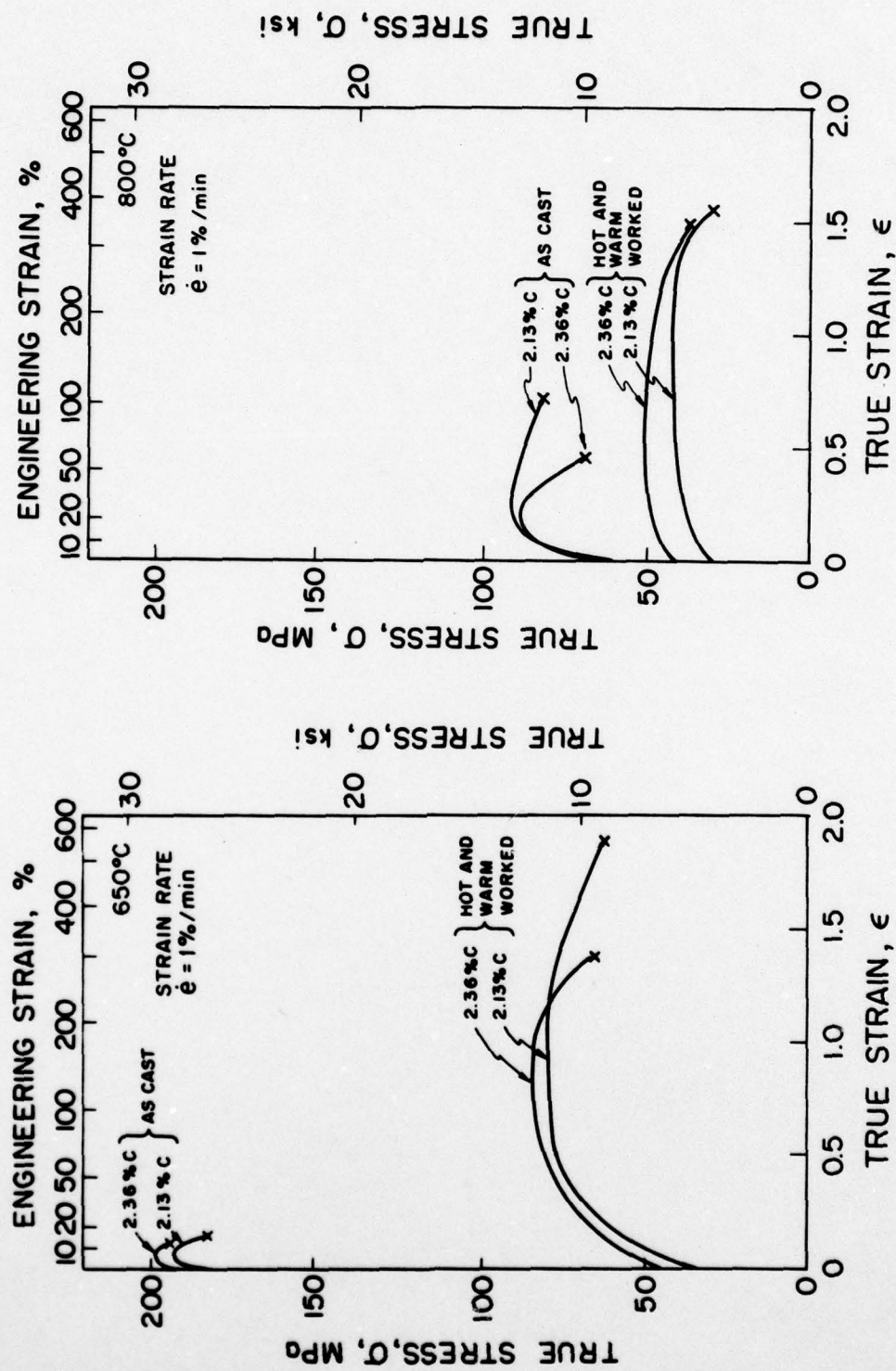
Figure 7. True stress–true strain curves for the 2.13%C and 2.36%C cast irons at a) 650°C in both the as-cast and hot-and-warm-worked conditions. and b) 800°C in both the as-cast and hot-and-warm-worked conditions.
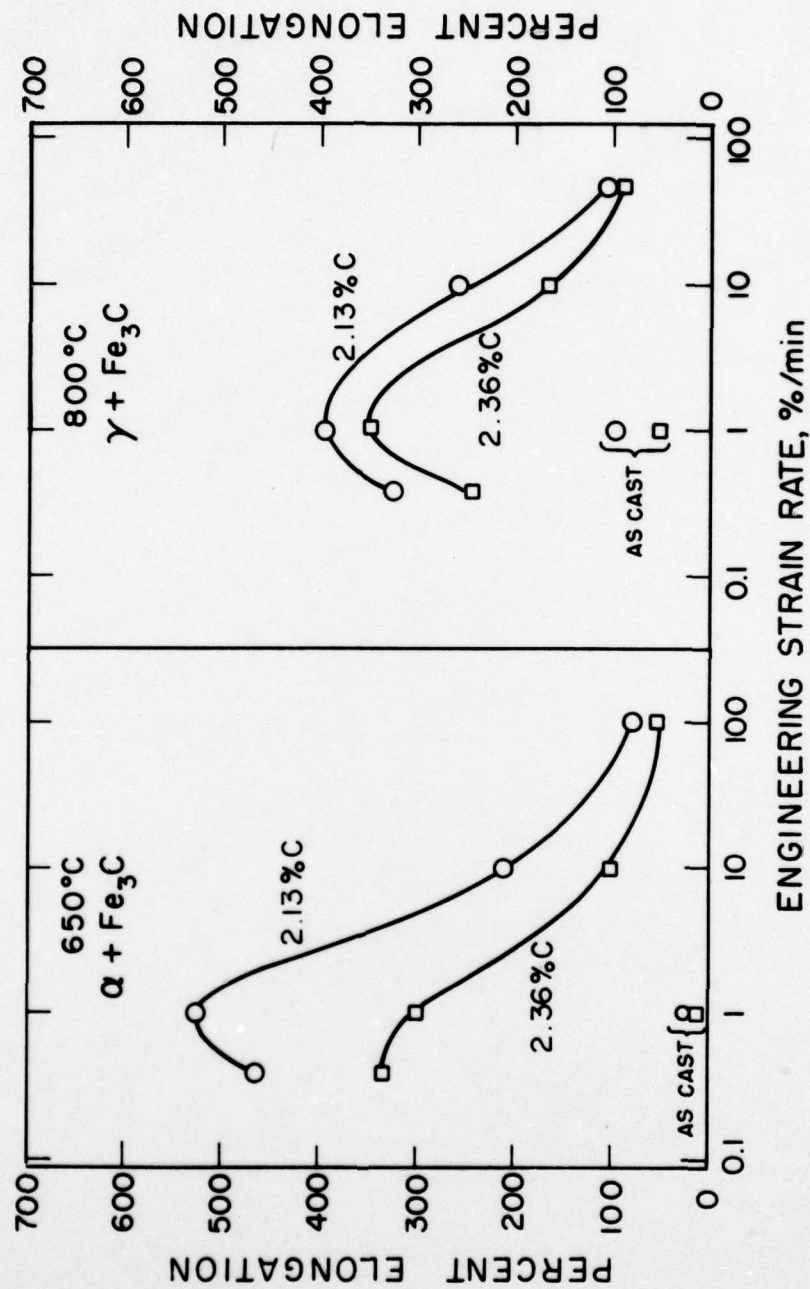
21.

Figure 8. The elongation-to-failure over a range of strain rates is shown at temperatures both above and below the $A_1$ for both the cast-irons. For comparison the values for the as-cast materials are also shown.
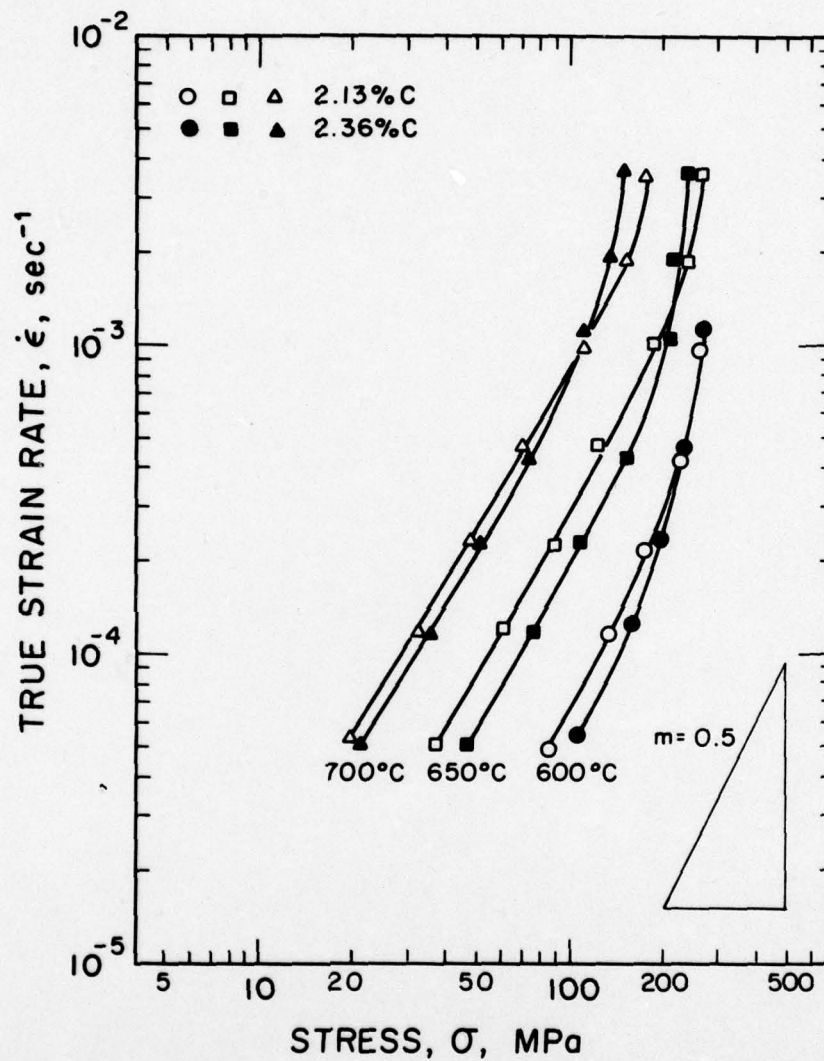
22.

TRUE STRAIN RATE, $\dot{\epsilon}$, sec$^{-1}$

$10^{-2}$

O □ △ 2.13%C
● ■ ▲ 2.36%C

$10^{-3}$

$10^{-4}$

m = 0.5

700°C   650°C   600°C

$10^{-5}$

5   10   20   50   100   200   500

STRESS, σ, MPa

Figure 9. Flow stress is measured at a number of imposed strain rates for both cast irons at temperatures below the $A_1$. Strain rate sensitivities on the order of 0.5 are found at low strain rates.
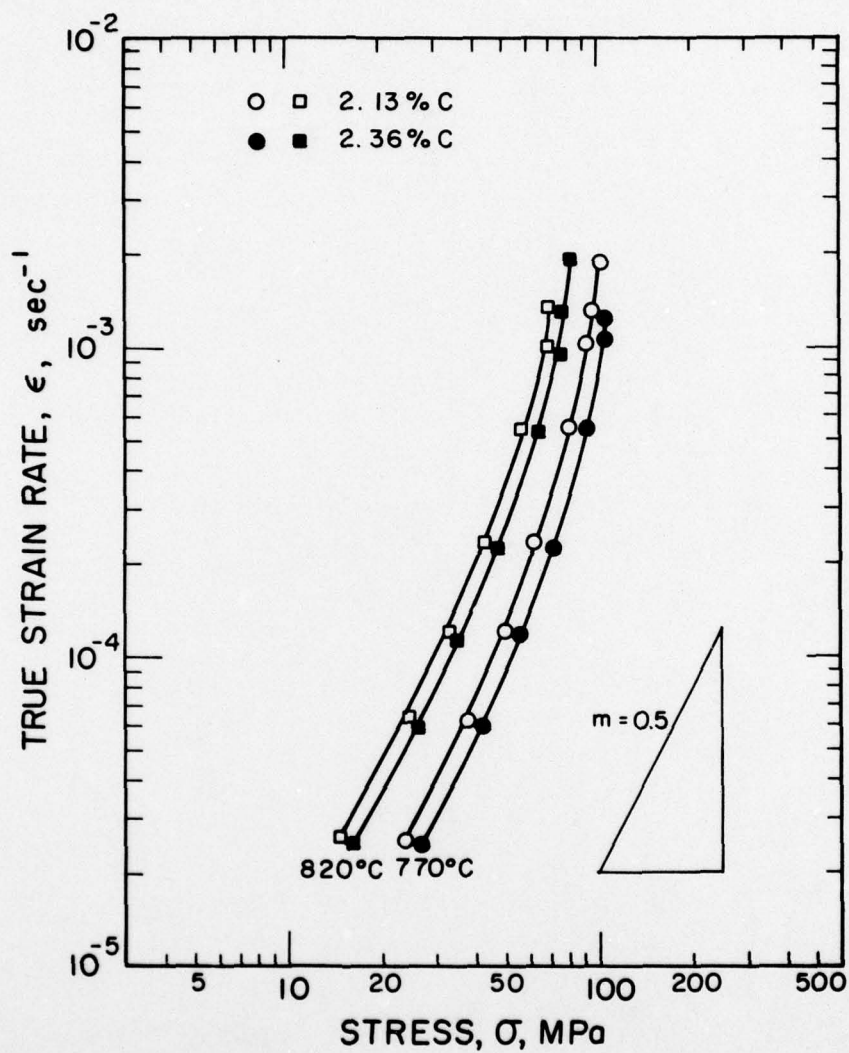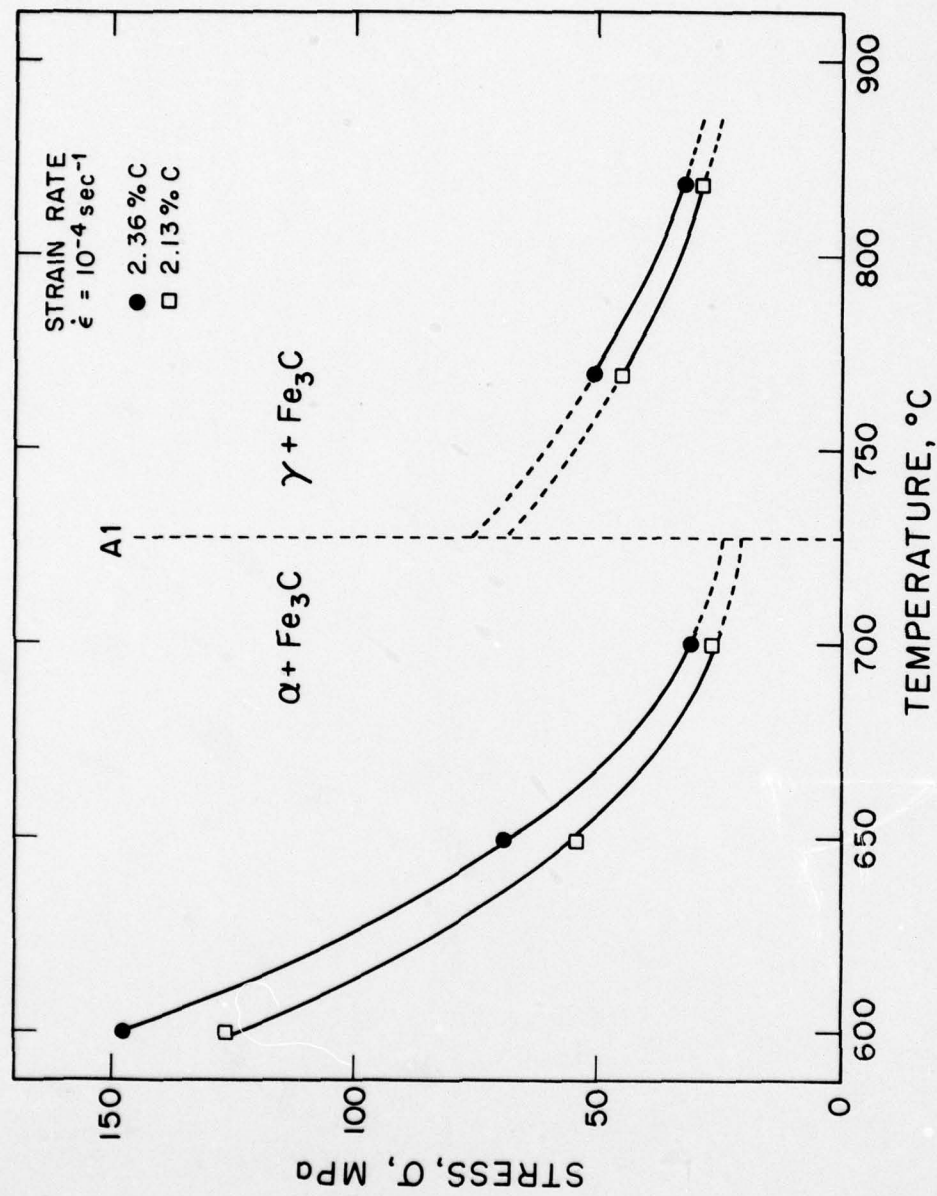
Figure 10. Flow stress is measured at a number of imposed strain rates for both the cast irons at temperatures above the $A_1$. Strain rate sensitivities on the order of 0.5 are found at low strain rates.

STRAIN RATE
$\dot{\epsilon} = 10^{-4}\ sec^{-1}$

● 2.36 %C
□ 2.13 %C

$\gamma + Fe_3C$

A1

$\alpha + Fe_3C$

STRESS, σ, MPa

150

100

50

0

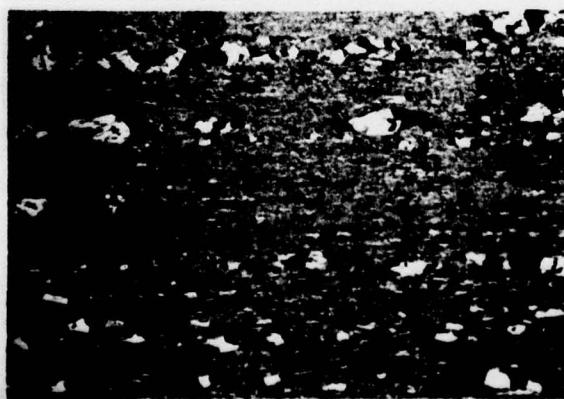600    650    700    750    800    900

TEMPERATURE, °C

Figure 11. Strength is plotted as a function of temperature for the 2.13%C and 2.36%C cast irons. Of both academic and technological interest is the low strengths of the cast irons at 700°C. The irons are weaker at 700°C than at 850°C.

25.
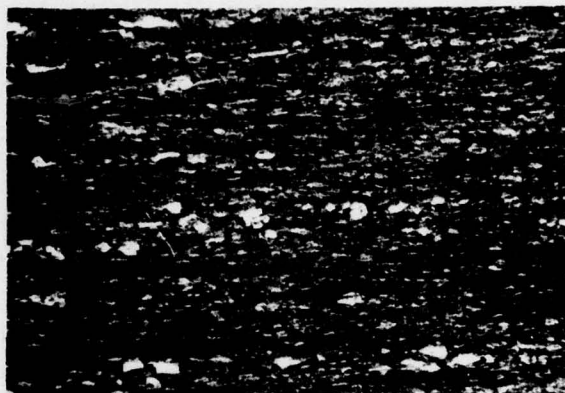
AS HOT AND
WARM WORKED

100μm

2.13%C
CAST IRON

TESTED IN
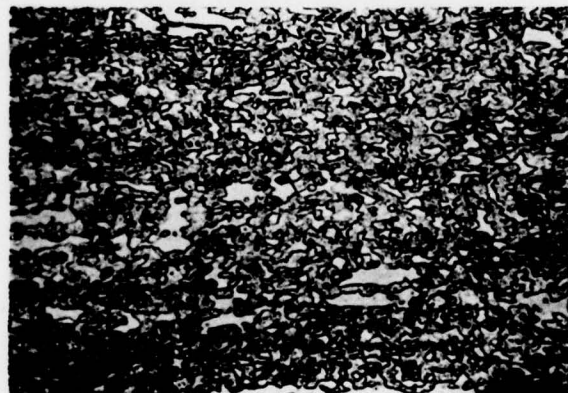TENSION AT
650°C TO
ABOUT 250%

100μm

Figure 12. Shown above are two optical micrographs of the hot forged, hot rolled and warm rolled cast iron containing 2.13%C. In the top micrograph the large undissolved cementite particles can be seen in a fine matrix of ferrite and cementite. Below is the same material after superplastic deformation at 650°C to about 250% at a strain rate of 1%/min. Cavitation can be seen to be exclusively associated with the large undissolved cementite particles.

2.13%C
CAST IRON

TESTED TO
200% IN
TENSION AT
650°C

MICROGRAPHS
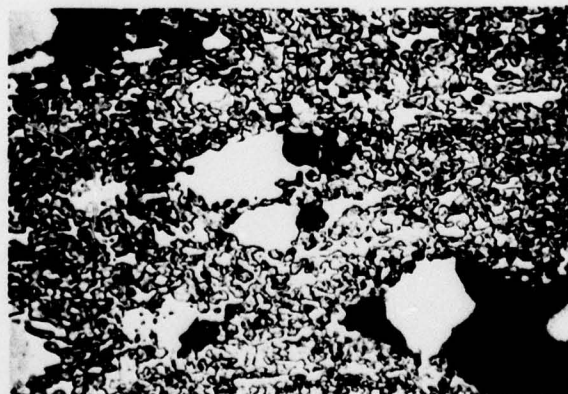TAKEN FROM
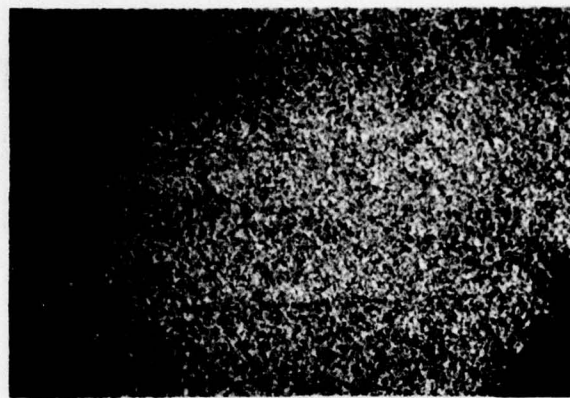ADJACENT
AREAS ON THE
GUAGE LENGTH

Figure 13. Shown above are further examples of the cavitation described in Figure 12.  As may be seen from the center micrograph, in areas free from carbides of > ~10μm in size, there is no cavitation.

1.6 % C

ULTRAHIGH
CARBON STEEL

TESTED TO
OVER 1100%
IN TENSION
AT 650°C

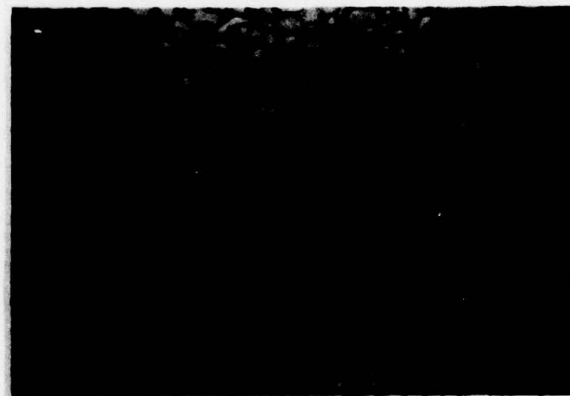100μm

MICROGRAPHS
TAKEN CLOSE
TO FRACTURE
SURFACE

10 μm

Figure 14. No cavitation is observed in an UHC steel tested to over
1100% in tension at 650°C.

28.

2.13% C

2.36% C

0          5          10 cm

Thermomechanically processed white cast irons
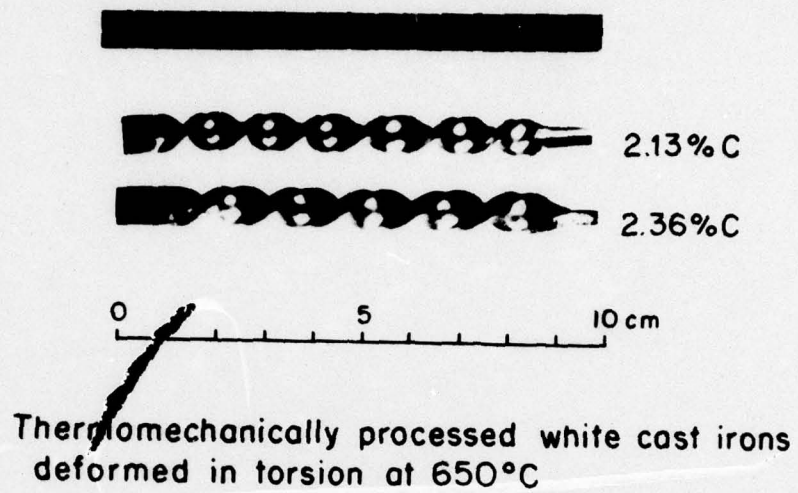deformed in torsion at 650°C

Figure 15. The white cast irons after thermomechanical processing deformed
in torsion at 650°C.  No cracking is visible in either iron after deformation.

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13. ABSTRACT

White cast irons are not normally considered malleable. Recent work at Stanford, however, has demonstrated that these materials can in fact be hot forged, hot rolled and warm rolled. Further, such a series of thermo-mechanical treatments refines the as-cast structure so as to impart unusual and highly beneficial properties to the cast irons. In particular, the worked, white cast irons are found to be superplastic and there is a great improvement in room temperature properties. This processing procedure may therefore have significant technological applications.

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| 14 KEY WORDS | LINK A | | LINK B | | LINK C | |
|---|---|---|---|---|---|---|
| | ROLE | WT | ROLE | WT | ROLE | WT |
| White cast iron Superplasticity Fine grain size | | | | | | |